DELIVERABLES REPORT

Multipurpose hemp for industrial bioproducts and biomass

(Ref n. 311849)

6.9 Report on the evaluation of a pilot-scale pultrusion testing device

August 2016

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1. Background and objectives

As a widely available renewable resource with low density and high stiffness, bast fibres have more and more been used to produce composite materials as a promising alternative to glass and petrochemical based fibres. Hemp can be considered as one of the oldest crops known to man, is a sustainable high yielding crop with advantageous environmental and agronomical characteristics and provides fibre of superior quality. Applied as reinforcement in composites materials, hemp fibre has been researched, for example, to produce car interior panels. In previous tasks of WP 6, profound analyses and investigations have been carried out with the focus on hemp fibres and the production to produce these fibres. Fibres have been evaluated and compared among hemp samples from different varieties and locations regarding the following properties: fibre length, fibre fineness, dislocations and mechanical properties. Within this task, the MultiHemp fibres were investigated as reinforcement for composite materials. Research on hemp fibre-reinforced composites is mainly focused on composite materials reinforced with short hemp and a low degree of fibre orientation. Evaluating these kinds of composite materials the reinforcing potential of the natural fibres can not be completely exploited. Composite structures reinforced with aligned long natural fibres/fibre bundles can obtain higher fibre volume fraction and offer higher mechanical strength. Within this MultiHemp WP activity, unidirectional composite samples with long hemp were produced by the pultrusion process investigated regarding their flexural properties.

2. Experimental details

2.1 Materials

Long hemp samples (hackled fibre bundles ca. 80 cm in length) of 14 varieties were prepared at UCSC. Each variety were cultivated in 4 blocks (B1, B2, B3, B4), block B3 was used to produce composite materials by the plutrusion process. The composites specimens from block B3 were tested at HSB and the fibre samples from the other three blocks were sent to KUL for the high-tech composite investigations (Task 5.1.1).

2.2 Composite manufacturing

A pilot-scale pultrusion device (compare figure 1) was built according to ISO 3597-1 at HSB, in order to produce the unidirectional composite samples. A standard procedure to produce the composite specimens was developed within the MultiHemp project. To produce composite samples with a desired fibre content the required amount of fibre bundles was weighted with a scale (Kern 440-35 M; D = 0.01 g; Kern & Sohn GmbH, Balingen, Germany). After drying for three houre at 105 °C in a drying cabinet the hackled hemp was impregnated for 10 minutes in a resin bath, where expoxy infusion resin RIM 135 and hardener RIMH 137i (supplied by Lange + Ritter

GmbH, Gerlingen, Germany) were mixed with a ratio of 10:3 by mass. Based on our preliminary investigations within the MultiHemp project and the development of the pultrusion procedure, a fibre content of 45% by mass (m%) can be achieved using bast fibres. A mass of 5.7 g hackled hemp is needed to produce a 35 cm long composite rod with a 6-mm diameter. After impregation, the fibre bundles are slowly pulled into a mould (hollow PTFE-cylinder; THOMAPLAST-Virginale-PTFE-Rohre-extrudiert, type: THOMAFLUOR-TU214-High Quality, Reichelt Chemietechnik GmbH + Co., Heidelberg, Germany). PTFE-tubes with different inner diameters are avaible, and can be cut to different length. In our work, tubes with 6-mm inner diameter were used (compare figure 2). The composite specimens were cured in the mould for 48 hours at ambient temperature, and then were demoulded. According to the testing procedure, the composites were cut into the required test specimen length of 120 mm with a band saw.

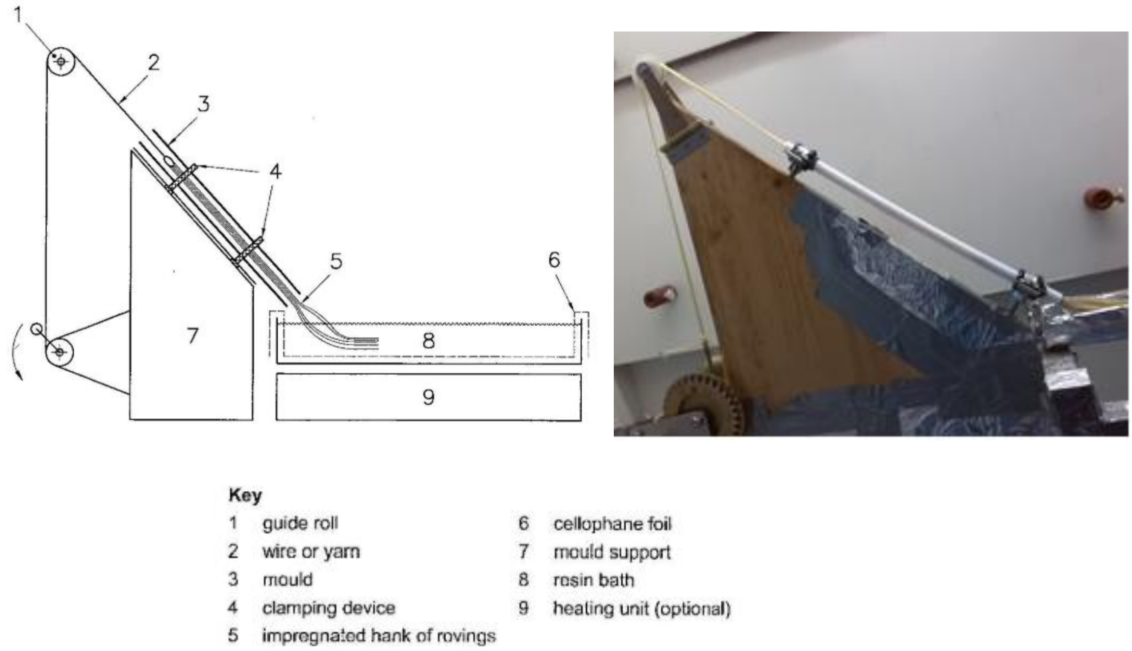


Figure 1: *Set-up of the pilot-scale pultrusion device.*

Figure 2: *Composite samples (35 cm long) from the pultrision process; left: hackled hemp in the PTFE mould; right: hemp fibre-reinforced composite rod.*

2.3 Flexural testing

For measuring the flexural properties a test set-up (figure 3), as described in DIN EN ISO 14125, was used with the following additional characteristics as specified in ISO 3597-2. A rod applicator (radius of waist: 5.6 mm) was applied, the radius of the suppoorts was 5 mm and the span between the supports was 96 mm. The testing device was a Zwick/Roell Z020 universal testing machine (Zwick GmbH and Co., Ulm, Germany) with a 20 kN load cell. A pre-load of 10 N was applied and the testing speed was 5 mm/min. Flexural modulus was calculated between 0.25 % to 0.5 % of flexural strain. The length of rod specimens was 120 mm, and for each sample 8 specimens were tested. Accroding to DIN EN ISO 291, prior to testing, the specimens were conditioned at 23 °C and 50 % relative humidity for 24 hours. For samples with no clear point of failure ("stepwise failure" compare figure 4; sample AGM-703 B3), a force value of 150 N was used as a stop criterion. If the force decreased under this limit after reaching the maximum force level the test stops automatically.

Figure 3: *Setup for bending tests*

3. Results and discussion

The composite samples of different hemp varieties show the similar failure behaviour in the bending test, and they collapsed with a sudden catastrophic drop in strength (compare figure 4; sample VDS-303 B3). The failure behaviour of the samples can also be identified in their fracture surfaces (see figure 5), and they failed due to tensile fracture at the outermost layer.

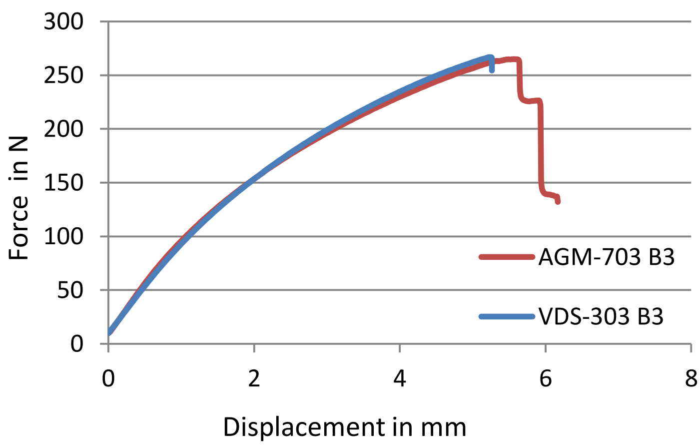


Figure 4: *Characteristic force–displacement curves of the flexural bending test*

Figure 5: *Characteristic damage at the tensile side of the test specimens after bending.*

The results of the bending test (flexural strength and modulus) of the 14 hemp composite samples are shown in Figure 6.

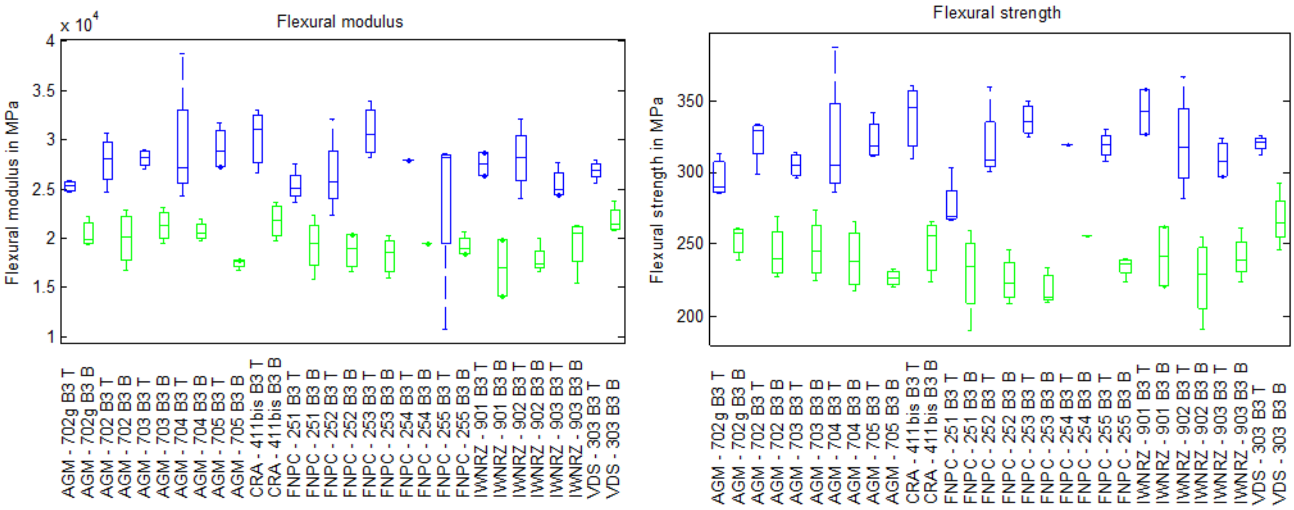


Figure 6: *Bending modulus and strength of the 14 samples (T: top specimen, B: bottom specimen).*

The two specimens, cut from the 35 cm long rod, contain different amounts of fibres because the fibre bundles from the hackled hemp used to produce the composite rods did not have an uniform longitudinal form and were thicker in the middle part. These differences can also be found and proved by density measurements (compare figure 7) and microsections of the composite specimens, which were mounted in resin (compare figure 8). In order to offer accurate and reliable results we did not cluster the samples which were cut from the top (T) and bottom (B) of the composite rods. For some varieties we found a high scattering of the mechanical properties and the densities in the top part as well as in the bottom part of the rod. The main reason for this is due to the uneven hackling process carried out manually at UCSC.

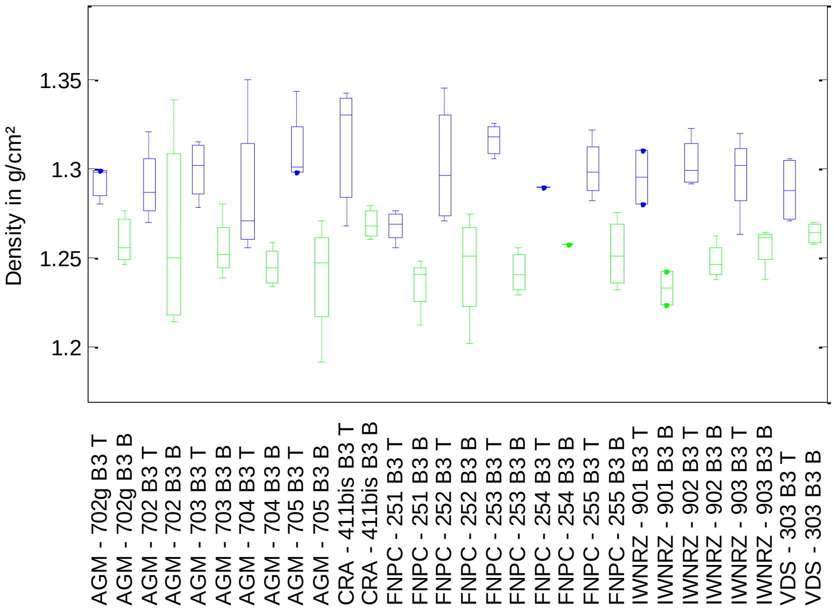
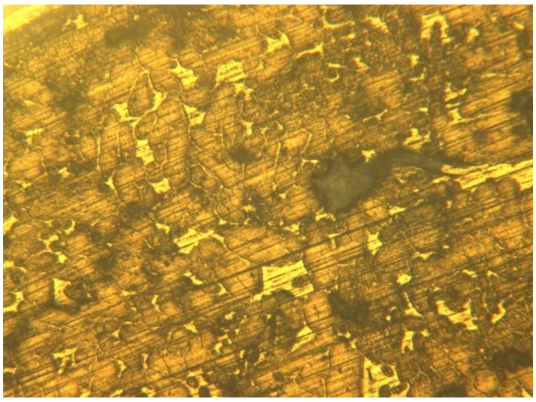


Figure 7: *Density of the top and bottom specimens of the 14 composite samples.*



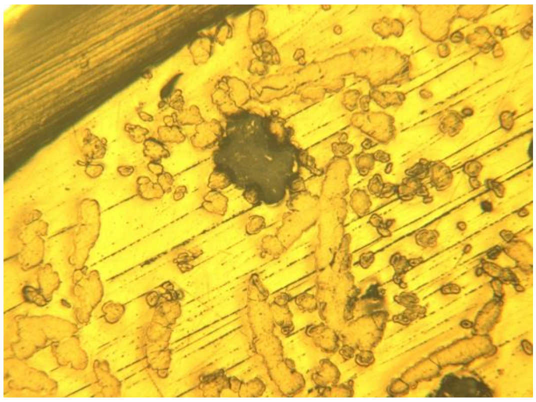


Figure 8: *Microscopic images of the mircosection of (i) left: the top part of the rod with a fibre content above 45 mass% (AGM-705 B3) and (ii) right: the bottom part of the rod with a fibre content below 30 mass% (AGM-705 B3).*

In order to find a solution to calculate the fibre content of the specimens after pultrusion in the top and bottom part, experiments have been carried out to determine the top and bottom portions of fibres in the different sections of the pultrusion mould. The results of this experiment are shown in Table 1. The measured data show a relatively large scattering for the different varieties and attempts to find mathematical correlations was not found to be possible.

Table 1: *Top and bottom portions of fibres in the mould after pultrusion of the hackled hemp without using resin.*

Fibre sample	mass in g at 105 °C	Top portion in g	Bottom portion in g	Top portion in %	Bottom portion in %	Top sample in v%	Bottom sample in v%
AGM-705-B3	5.25	2.04	1.68	38.9	32.0	40.6	33.5
FNPC-255-B3	5.25	2.12	1.75	40.4	33.3	42.2	34.8
IWNRZ_902-B3	5.20	1.97	1.71	37.9	32.9	39.2	34.0
IWNRZ-903-B3	5.28	2.18	1.82	41.3	34.5	43.4	36.2
VDS-303-B3	5.28	2.44	1.87	46.2	35.4	48.6	37.2
CRA-411-B3	5.24	2.35	1.65	44.8	31.5	46.8	32.9

A first approach would be to simple modelling (see Eq. 1) the fibre content by volume (***x*** in *v%*) using the density of the fibre ρ_f, matrix ρ_m and specimen ρ_c

$$\rho_c = \rho_f \cdot \frac{x}{100} + \rho_m \cdot \left(1 - \frac{x}{100}\right) \Longrightarrow x = \frac{\rho_c - \rho_m}{\rho_f - \rho_m} \times 100 \qquad \text{(Eq. 1)}$$

With a density $\rho_f = 1{,}48\ g/m^3$ for the hemp fibre and $\rho_m = 1{,}15\ g/m^3$ for the matrix used the fibre content of the 14 composite samples were calculated and are shown in figure 9.

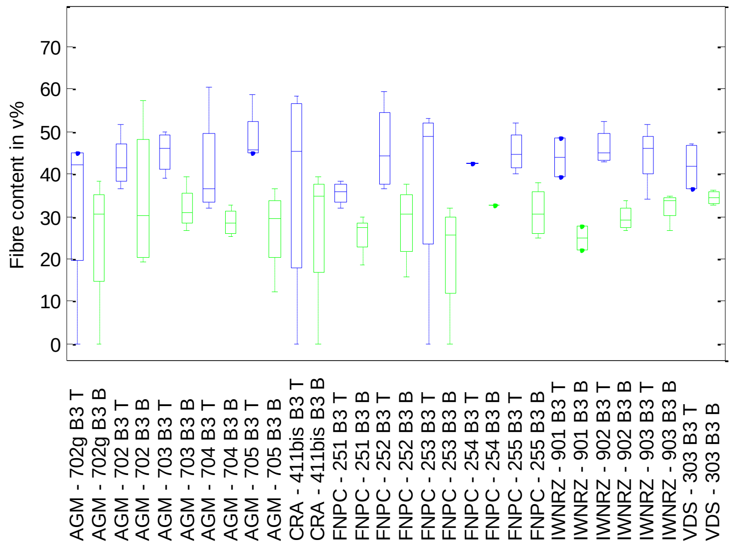


Figure 9: *Calculated fibre volume content of the 14 composite samples.*

The next step in our data evaluation is the development of a mathematical model which enables us to evaluate the material properties independently of their firbe content, because the properties of composites are strongly affected by fibre content (see figure 10).

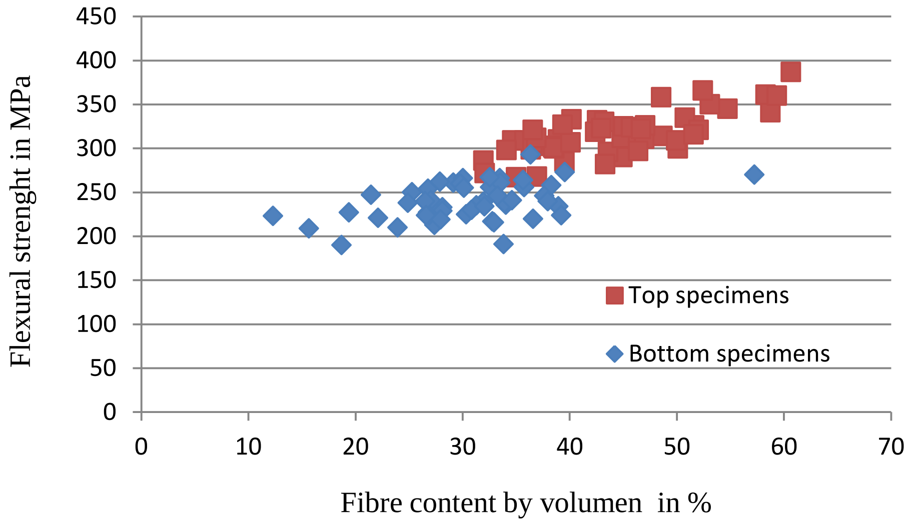


Figure 10: *Correlation between fibre content by volumen and flexural strength.*

Baets & Verpoest (2012) developed the "flax playground" to show the range of strength and stiffness that can be achieved by different flax composites (see Figure 11). The mechanical properties that can be achieved with a certain processing technique at a set fibre content define the suitability for an application.

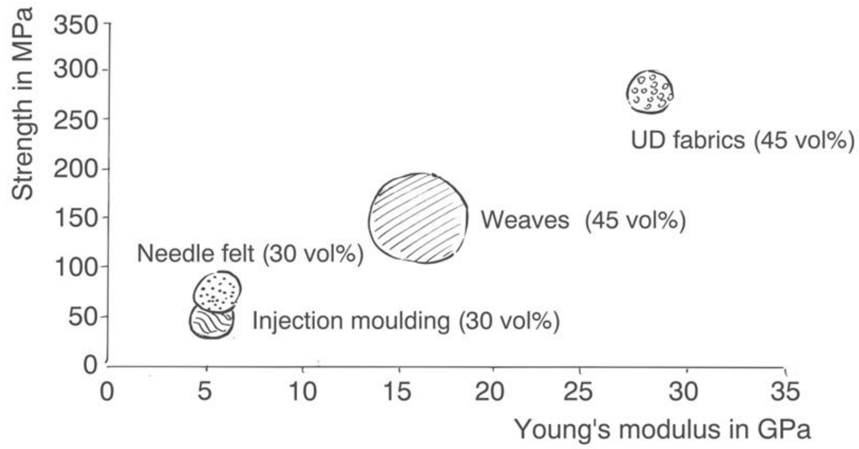


Figure 11: *Performance playground of flax composites (adapted from Verpoest & Baets, 2012 in Müssig & Haag, 2015)*

The highest strength and modulus values can be achieved, for example, by pultrusion with high fibre content, high orientation and low damage of the fibre bundles. Our results within the MultiHemp project are very promising, because our hemp UD pultrusion composites reach properties comparable to high-quality flax UD composites (compare figure 11).

References

Müssig, J. & Haag, K. (2015): *2 - The use of flax fibres as reinforcements in composites.* In: Faruk, O. & Sain, M. (Eds.): Biofiber Reinforcements in Composite Materials, Woodhead Publishing, 2015, 35 – 85

Verpoest, I. & Baets, J. (2012): *Flax and hemp fibre composites.* In: JEC composites conference in partnership with CELC (organizers): Flax and Hemp Fibres: A Natural Solution for the Composite Industry – High-performance operational green-chemistry solutions (Leuven, Belgium, 4 December 2012), Paris: JEC and CELC, CD-ROM, Conference Proceedings, 17 pp.